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14. ABSTRACT

We have focused on developing innovative ways of thermal management and also enhancement of thermoelectric performance through thermal conductivity control. We have carried out various high temperature synthesis or relatively low temperature synthesis utilizing flux growth methods and also bottom-up nanosheet fabrication for the materials studied in the work. Studies have included: $RAlB_4$ crystals, AlB_2, bismuth telluride-type nanosheets, rare earth borocarbonitrides, B6S1-x compound, and carrier-doped chalcopyrite. As a striking byproduct of this work, we investigated the properties of a complex phase chalcogenide compound, and although thermoelectric properties were not particularly good, an impactful scientific result was achieved, in the elucidation of the origin of the superconducting phase. Overall, we propose that magnetic semiconductors may be another new frontier for obtaining high performing thermoelectrics.

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Final Report for AOARD Grant 1214046

"Utilizing interfaces for nano- and micro-scale control of thermal conductivity"

Date

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Abstract: We have focused on developing innovative ways of thermal management and also enhancement of thermoelectric performance through thermal conductivity control. With the aim of utilizing building ("tiling") defects which have recently been discovered in layered materials, we have established flux growth methods to reliably synthesize and control creation of building defects in $RAlB_4$ crystals. Time domain thermoreflectance measurements were carried out on microcrystals and it was discovered that the building defects are a mechanism to substantially reduce thermal conductivity by ~30% in crystals examined. To enhance tools to investigate the building defects which strongly affect thermal conductivity further, HAADF-STEM was successfully demonstrated as a powerful method to visualize the small planar-domains. Furthermore, a striking contrast with the thermal transport of carbon/graphene materials was discovered in that AlB_2, which structure is like [B]-graphene sheets sandwiching Al, shows higher thermal conductivity perpendicular to the planes compared to the in-plane [B]-graphene direction. This is intuitively completely opposite of that would be expected for graphene and illustrates the particular bonding of boron. In an alternate method to control thermal conductivity, through a bottom-up method, we have been able to fabricate nanosheets of a thermoelectric material, copper telluride, and achieved a striking low lattice thermal conductivity (lower than amorphous silica glass) without incurring a large penalty in the electrical conductivity. We have also synthesized bismuth telluride-type nanosheets, evaluated the properties and are in the process of modifying the balance of thermal conductivity and electrical conductivity, since bismuth telluride-type materials are the champion thermoelectric materials at room temperature. Compositional changes during the synthesis methods of nanosheets were shown to strongly affect the properties. The nanosheet approach to introduce nanostructuring of materials is revealed to be a powerful method for controlling thermal conductivity going forward. An easy mechanical method to strongly control thermal conductivity was demonstrated whereby a record ultra-low thermal conductivity was achieved in bismuth telluride by room temperature deformation. We have also proceeded with development of an advanced revolutionary method on utilizing nanoscale probes in TEM to achieve nanoscale resolution in evaluating thermal conductivity/thermal resistance on the interfaces of hybrid/composite materials matching theoretical predictions. The thermal resistance of the interface between the resin and fillers in a heat sink composite material was successfully evaluated. Several promising thermoelectric materials were also realized in the course of testing the symmetry mismatch effect which we have proposed may be a novel thermal conductivity effect in icosahedral cluster compounds. A striking enhancement of ~40 times in the ZT was obtained for the samarium phase of RB_{66}, the first actual phonon glass electron crystal (PGEC). The origin of the n-type anomalous behavior in the rare earth borocarbonitrides, which were discovered as long-awaited n-type counterparts to boron carbide, was also elucidated. We have also recently been successful in varying excellent (absolute Seebeck coefficients >200 μ V/K) p-type or n-type characteristics in an aluminoboride $Y_xAl_yB_{14}$ (x ~ 0.57) by controlling the y occupancy of the Al site. Since p-n control of materials has traditionally been one of the large obstacles to developing applicable thermoelectric materials, this discovery of such control in a material with same basic crystal structure framework (therefore good matching) and no necessity for doping of foreign elements (therefore no migration problems) is very striking. In further development, addition of Al as a sintering element was found to enhance the thermoelectric performance of these promising materials by 50%. In lowering the thermal

conductivity, conventional ballmilling was found to not be effective, and a novel synthesis method to fabricate the boron framework first, actually yielded results, since due to shorter heating time at lower temperature, the grain sizes was successfully decreased, decreasing thermal conductivity. In other results, by investigation of the insertion of sulpher atoms into voids of the boron cluster we have synthesized a possible replacement material of boron carbide (one of the few thermoelectric materials besides Bi_2Te_3 with a history of commercialization), which can be prepared at much lower temperatures. With ramifications for many thermoelectric materials, we have discovered a hybrid effect by which the electrical conductivity and Seebeck coefficient could be simultaneously significantly enhanced, breaking the traditional tradeoff relation, which has been one of the bottlenecks impeding thermoelectric performance. We have also discovered unexpectedly high thermoelectric power factors in carrier-doped chalcopyrite. The base material is actually a mineral and composed of common, inexpensive elements. Further development these compounds are phase stable to 700 K, and maintain a large power factor of 1 mW/K^{2}m in the temperature range of 400 K to 600 K. As a striking byproduct of this work, we investigated the properties of a complex phase chalcogenide compound, and although thermoelectric properties were not particularly good, an impactful scientific result was achieved, in the elucidation of the origin of the superconducting phase. Overall, we propose that magnetic semiconductors may be another new frontier for obtaining high performing thermoelectrics.

Introduction: Approximately two thirds of all primary energy (fossil fuels, etc.) being consumed in the world, sadly turns out to be unutilized, with much of the waste being in the form of heat. Developing innovative ways of thermal management and also enhancement of thermoelectric performance through thermal conductivity control will be one of the most important technological issues going forward. However, while control over electrical carriers has reached state of the art level in many technologies, a similar control and understanding over phonons in complex systems is still lacking, and development of the fundamental science needs to be accelerated.

Experiment: We have carried out various high temperature synthesis or relatively low temperature synthesis utilizing flux growth methods and also bottom-up nanosheet fabrication for the materials studied in the work.

Results and Discussion: Describe significant experimental and/or theoretical research advances or findings and their significance to the field and what work may be performed in the future as a follow on project. Fellow researchers will be interested to know what impact this research has on your particular field of science.

(1) In searching for novel methods to control thermal conductivity, with the aim of utilizing ubiquitous building ("tiling") defects which have recently been discovered in layered materials, we have established flux growth methods to reliably synthesize and control creation of building defects in $RAlB_4$ crystals. Time domain thermoreflectance measurements were carried out on microcrystals and it was discovered that the subtle nanoscale building defects are a mechanism to substantially reduce thermal conductivity by ~30% in crystals examined.
(2) A striking contrast with thermal transport in carbon/graphene materials was discovered in that AlB_2, which structure is like [B]-graphene sheets sandwiching Al, shows higher thermal conductivity perpendicular to the planes compared to the in-plane [B]-graphene direction. This is intuitively completely opposite of that would be expected for graphene and illustrates the particular bonding of boron.
(3) To enhance tools to investigate the building defects which strongly affect thermal conductivity further, HAADF-STEM was successfully demonstrated as a powerful method to visualize the small planar-domains.
(4) In further efforts to utilize nanoscale interfaces to control thermal conductivity, we have been able to achieve striking effects with different methods compared to the conventional ballmilling methods in creating nanoscale interfaces. One is a bottom-up method, where we have been able to fabricate nanosheets of a thermoelectric material, copper telluride, and achieved a striking low lattice thermal conductivity (lower than amorphous silica glass) without incurring a large penalty in the electrical conductivity. We have also synthesized bismuth telluride-type nanosheets, evaluated the properties and are in the process of modifying the balance of thermal conductivity

and electrical conductivity, since bismuth telluride-type materials are the champion thermoelectric materials at room temperature. Compositional changes during the synthesis methods of nanosheets were shown to strongly affect the properties.

(5) An easy mechanical method was demonstrated (in contrast to time&energy consuming ballmilling), whereby with room temperature deformation we were able to obtain ultra-low thermal conductivity in bismuth telluride.

(6) Related to the above work, having a probe to evaluate the thermal conductivity on the nanoscale of desired locations in a material has been a challenging problem. We have initiated work on developing a novel method to directly evaluate nanoscale thermal conductivity. Namely, we have initiated development of an advanced revolutionary method, and made the first successful measurements on utilizing nanoscale probes in TEM to achieve nanoscale resolution in evaluating thermal conductivity/thermal resistance on the interfaces of hybrid/composite materials. The thermal resistance of the interface between the resin and fillers in a heat sink composite material was successfully evaluated and matched theoretical predictions.

(7) Several promising thermoelectric materials were also realized in the course of testing the symmetry mismatch effect which we have proposed may be a novel thermal conductivity effect in icosahedral cluster compounds. The RB_{66} system is an interesting one, since it can be said to be the actual first phonon glass electron crystal (PGEC). SmB_{66} single crystals were grown and as a result, a striking enhancement of ~40 times in the ZT was obtained for the samarium phase compared to other rare earths. The origin of the n-type anomalous behavior in the rare earth borocarbonitrides was also elucidated. The rare earth borocarbonitrides are of interest as the long-awaited n-type counterparts to boron carbide. We have also recently been successful in varying excellent (absolute Seebeck coefficients >200 μ V/K) p-type or n-type characteristics in an aluminoboride $Y_xAl_yB_{14}$ (x ~ 0.57) by controlling the y occupancy of the Al site. Since p-n control of materials has traditionally been one of the large obstacles to developing applicable thermoelectric materials, this discovery of such control in a material with same basic crystal structure framework (therefore good matching) and no necessity for doping of foreign elements (therefore no migration problems) is very striking. In further developments, addition of Al as a sintering element was found to enhance the thermoelectric performance of these promising materials by 50%. Ballmilling and spark plasma sintering (SPS) processes were investigated to try to lower the thermal conductivity and enhance the thermoelectric performance. Mechanical grinding using ball milling with Si_3N_4 pots and balls was found not to be an efficient way to decrease the grain size because of contamination of Si_3N_4. In contrast "$Y_xAl_yB_{14}$ via $Y_{0\ 56}B_{14}$" samples were successfully synthesized. Through the synthesis of $Y_{0\ 56}B_{14}$, the boron network structure was first formed. Afterward, $Y_xAl_yB_{14}$ was obtained by adding Al in the boron network structure through a heat treatment. Due to shorter heating time at lower temperature, the grain sizes were discovered to be smaller than that of Al flux method. The decrease of grain size was found to be beneficial for the densification of $Y_xAl_yB_{14}$ and the decrease of its thermal conductivity.

(8) By investigation of the insertion of sulpher atoms into voids of the boron cluster we have synthesized a possible replacement material of boron carbide (one of the few thermoelectric materials besides Bi_2Te_3 with a history of commercialization which can be prepared at much lower temperatures. been successful to synthesize the B6S1-x compound which was found to have S-S dumbbells at relatively low temperatures. The TE is comparable to boron carbide one of the few TE materials previously commercialized. Boron sulfide can potentially be a replacement TE materials for boron carbide able to be prepared with much milder synthesis conditions.

(9) We have discovered a striking hybrid effect on doping particular kinds of transition metals (TM) combined with a heat treatment process. In TE the electrical conductivity and Seebeck coefficients are in a tradeoff relationship and normally it is not possible to increase both. We have discovered strikingly, a 2 order enhancement in electrical conductivity coupled with a 220% increase in Seebeck coefficients, shattering the conventional tradeoff. We surmise that with our process we achieved a hybrid effect where microscopically electrical paths could be created in the material while simultaneously the bulk base material's electronic structure was modified by partial intrinsic TM doping which modified the electronic structure to increase the Seebeck. (Note: in this case, the bulk base material's electrical conductivity is most likely lowered, but successfully bypassed by the electrical paths).

(10) We have discovered unexpectedly high thermoelectric power factors in carrier-doped chalcopyrite. The base material is actually a mineral and composed of common, inexpensive

elements. Despite this, we have achieved a power factor which approaches that of the famous TE material bismuth telluride, which are at opposite ends of the Clark number graph. We have concluded that the TE enhancement is due to magnetic interactions and propose that magnetic semiconductors may be the new frontier for obtaining high performing TE. This result was selected as a spotlight of APEX. Further, development has shown regarding the phase stability of these compounds they are stable to 700 K, and maintain a large power factor of 1 mW/K^{2}m in the temperature range of 400 K to 600 K. As a byproduct of this work, we investigated the properties of a complex chalcogenide system, and although thermoelectric properties were not particularly good, the origin of the superconducting phase was elucidated. Even though this result is a byproduct not initially expected in the plan, it is a significant scientific result and led to a high impact paper in *Nature Communications*.

List of Publications and Significant Collaborations that resulted from your AOARD supported project: In standard format showing authors, title, journal, issue, pages, and date, for each category list the following:

a) papers published in peer-reviewed journals,

1. X. J. Wang, T. Mori, I. Kuzmych-Ianchuk, Y. Michiue, K. Yubuta, T. Shishido, Y. Grin, S. Okada, and D. G. Cahill, "Thermal Conductivity of Layered Borides: the Effect of Building Defects on the Thermal Conductivity of TmAlB4 and the Anisotropic Thermal Conductivity of AlB2" APL Materials 2, 046113-1 046113-6 (2014). http://dx.doi.org/10.1063/1.4871797
2. K. Yubuta, T. Mori, S. Okada, Y. Prots, H. Borrmann, Y. Grin, and T. Shishido, "High-Resolution Electron Microscopy and X-ray Diffraction Study of Intergrowth-Structures in α- and β-Type $YbAlB_4$ Single Crystals", Philosophical Magazine, 93 (2013) 1054-1064. DOI: 10.1080/14786435.2012.741727
3. R. Ang, C. L. Chen, Z. C. Wang*, J. Tang, N. Liu, Y. Liu, W. J. Lu, Y. P. Sun, T. Mori* & Y. Ikuhara, "Atomistic Origin of Ordered Superstructure Induced Superconductivity in Layered Chalcogenides" Nature Commun. 6, 6091 (2015). DOI: 10.1038/ncomms7091
4. C. Nethravathi, R. Rajamathi, M. Rajamathi, R. Maki, T. Mori, D. Golberg, Y. Bando, "Synthesis and thermoelectric behaviour of copper telluride nanosheets" Journal of Materials Chemistry A 2 (2014) 985-990 DOI:10.1039/c3ta12877f
5. S. Grasso, N. Tsujii, Q. Jiang, J. Khaliq, S. Maruyama, M. Miranda, K. Simpson, T. Mori, M. J. Reece, "Ultra low thermal conductivity of disordered layered p-type bismuth telluride" , Journal of Material Chemistry C (2013), 1, 2362-2367. DOI: 10.1039/C3TC30152D
6. T. Mori, T. Nishimura, W. Schnelle, U. Burkhardt, and Y. Grin, "The origin of the n-type behavior in rare earth borocarbide $Y_{1-x}B_{28.5}C_4$", Dalton Trans., 43, 15048-15054 (2014). DOI: 10.1039/C4DT01303D
7. S. Maruyama, A. Prytuliak, Y. Miyazaki, K. Hayashi, T. Kajitani, and T. Mori, "Al insertion and additive effects on the thermoelectric properties of yttrium boride", J. Appl. Phys. 115, 123702 (2014). http://dx.doi.org/10.1063/1.4869131
8. S. Maruyama, T. Nishimura, Y. Miyazaki, K. Hayashi, T. Kajitani, and T. Mori, "Microstructure and thermoelectric properties of YxAlyB14 samples fabricated through the spark plasma sintering", Mater Renew Sustain Energy, 3, 31-1 31-6 (2014). DOI: 10.1007/s40243-014-0031-8
9. O. Sologub, Y. Matsushita and T. Mori, "α-Rhombohedral Boron Related Compound with Sulfur: Synthesis, Structure and Thermoelectric Properties", Scripta Materialia 68 (2013) pp. 288-291. 10.1016/j.scriptamat.2012.10.044
10. A. Prytuliak, S.Maruyama, and T. Mori, "Anomalous effect of vanadium boride seeding on thermoelectric properties of $YB_{22}C_2N$", Materials Research Bulletin, 48 (2013), pp. 1972-1977. 10.1016/j materresbull.2013.01.043
11. N. Tsujii and T. Mori, "High Thermoelectric Power Factor in a Carrier-Doped Magnetic Semiconductor $CuFeS_2$", Applied Physics Express, 6 (2013) 043001 1-4. Selected as a SPOTLIGHT http://dx.doi.org/10.7567/APEX.6.043001
12. N. Tsujii, T. Mori, and Y. Isoda, "Phase Stability and Thermoelectric Properties of $CuFeS_2$ -based Magnetic Semiconductor", Journal of Electronic Materials, 43, 2371-2375 (2014).
13. K. Yubuta, T. Mori, S. Okada, Y. Prots, H. Borrmann, Y. Grin, and T. Shishido, "High-Resolution Electron Microscopy and X-ray Diffraction Study of Intergrowth-Structures in α- and β-Type

YbAlB4 Single Crystals”, Philosophical Magazine, 93 (2013) 1054-1064.

14. S. Grasso, N. Tsujii, Q. Jiang, J. Khaliq, S. Maruyama, M. Miranda, K. Simpson, T. Mori, M. J. Reece, “Ultra low thermal conductivity of disordered layered p-type bismuth telluride” , Journal of Material Chemistry C, 1 (2013) 2362-2367.
15. O. Sologub and T. Mori, “Structural and thermoelectric properties of Y1-xB22+yC2-yN”, Journal of Physics and Chemistry of Solids 74 (2013) 1109–1114.
16. A. Prytuliak, S.Maruyama, and T. Mori, “Anomalous effect of vanadium boride seeding on thermoelectric properties of YB22C2N”, Materials Research Bulletin, 48 (2013) 1972-1977.
17. O. Sologub, Y. Michiue and T. Mori, “Boron carbide, B13-xC2-y (x = 0.12, y = 0.01)”, Acta Cryst. E68, i67 (2012).
18. O. Sologub, Y. Matsushita and T. Mori, “α-Rhombohedral Boron Related Compound with Sulfur: Synthesis, Structure and Thermoelectric Properties”, Scripta Materialia 68 (2013) 288-291.
19. N. Tsujii and T. Mori, “High Thermoelectric Power Factor in a Carrier-Doped Magnetic Semiconductor CuFeS2”, Applied Physics Express, 6 (2013) 043001 1-4. Selected as SPOTLIGHTS
20. T. Mori, R. Sahara, Y. Kawazoe, K. Yubuta, T. Shishido, and Y. Grin, “Strong magnetic coupling in a magnetically dilute f-electron insulator; a dysprosium boron-cluster compound”, Journal of Applied Physics 113, 17E156 (2013).

Invited Lectures only

1. Takao Mori, Annual Meeting of the Chemistry Society of Japan, “Utilizing low dimensional nanosheets and building defects to control thermal conductivity and enhance thermoelectric properties” [in Japanese], Tokyo, Japan, March 26, 2015.
2. Takao Mori, INC10 (The Tenth International Nanotechnology Conference on Communicati on and Cooperation) “Key issues and developments in thermoelectrics and thermal manage ment technology”, Gaithersburg, Maryland (NIST), May 13-15, 2014.
3. Takao Mori, Gordon Research Conference in Solid State Chemistry, “Development of nov el borides, silicides, sulfides, and nitrides as effective thermoelectric materials”, Colby-Saw yer College, July 27-August 1, 2014.
4. Takao Mori, CSC2014 (97th Canadian Chemistry Conference), “Utilizing network-like stru ctures to develop novel functions (magnetism and thermoelectrics) in inorganic compounds ", Vancouver, June 1-5, 2014.
5. Takao Mori, ISRS-18 (18th International Symposium on the Reactivity of Solids), “Development of novel borides, silicides, sulfides, and oxides, as effective thermoelectric materials”, St. Petersburg, June 9-13, 2014
6. Takao Mori, TMS2014, “Development of Novel Refractory Compounds as Thermoelectric Materials”, San Diego, February 19 (February 16-20), 2014.
7. Takao Mori, PACRIM10, “Development of novel borides, silicides, and oxides, as effective high temperature thermoelectric materials”, Coronado, June 4 (June 2-7), 2013.
8. Takao Mori, 2nd Dresden Conference: Energy in Future, “Development of Thermoelectrics in Japan”, Dresden, May 28 (May 28-29), 2013.
9. Takao Mori, TEP-CH 2013: Synthesis and Function of Thermoelectric Materials, “Development of High Temperature Thermoelectric Materials via Control of Network Structure”, Empa, Switzerland, September 17 (September 17-19), 2013.
10. Takao Mori, MRS Spring Meeting 2012, “Nanostructured Borides and Perspectives of High Temperature Thermoelectric Materials,” San Francisco, USA, April 13 (April 9~13), 2012.
11. Takao Mori, 3rd IAV Conference: Thermoelectrics, “Development of novel silicides, borides and oxides, as effective thermoelectric materials”, Berlin, November 22 (November 21–23), 2012.
12. Takao Mori, ICEAN2012 (International Conference on Emerging Advanced Nanomaterials – 2012), “Development of High Temperature Thermoelectric Materials; Doping Effects in Boron Cluster Compounds”, Brisbane, Australia, October 22 (October 22-25), 2012.
13. Takao Mori, MRS Spring Meeting 2012, “Nanostructured Borides and Perspectives of High Temperature Thermoelectric Materials”, San Francisco, USA, April 13 (April 9~13), 2012
14. Takao Mori, 3rd IAV Conference: Thermoelectrics, “Development of novel silicides, borides and oxides, as effective thermoelectric materials”, Berlin, November 22 (November 21–23), 2012.
15. Takao Mori, ICEAN2012 (International Conference on Emerging Advanced Nanomaterials –

2012), “Development of High Temperature Thermoelectric Materials; Doping Effects in Boron Cluster Compounds”, Brisbane, Australia, October 22 (October 22-25), 2012.

16. Takao Mori, The Japan institute of Electronics Packaging, Environment-conscious design technology study group, 2nd public seminar of FY2012, “Research and development of thermoelectrics materials for energy harvest and their newest trend” [in Japanese], Tokyo, Japan, January 23, 2013
17. Takao Mori, Symposium on thermal physics of environment and energy materials, “Finding a mechanism of low thermal conductivity for development of advanced thermoelectric materials” [in Japanese], Tokyo, Japan, November 20, 2012

e) manuscripts submitted but not yet published, and

1. Alif Sussardi, Takaho Tanaka, A. Ullah Khan, Louis Schlapbach, and Takao Mori, “Enhanced thermoelectric properties of samarium boride”, submitted to Journal of Materiomics.

2. N. Kawamoto, Y. Kakefuda, T. Mori, K. Hirose, M. Mitome, Y. Bando, and D. Golberg, “In situ TEM thermal resistivity analysis within a microscale composite sample by a piezo-driven nanothermocouple”, submitted to Nanotechnology.

f) provide a list any interactions with industry or with Air Force Research Laboratory scientists or significant collaborations that resulted from this work.